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DECOMPOSITION AND DEFLAGRATION OF AMMONIUM PERCHLORATE

by

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and

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ABSTRACT. This report contains studies of the following behavior of pure single crystals of ammonium perchlorate: decomposition in vacuum, high heating rate decomposition at atmospheric pressure, self-deflagration in nitrogen atmospheres of $275 \leq p \leq 5000$ psia and deflagration in methane atmospheres of $15 \leq p \leq 200$ psia.

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FOREWORD

This report summarizes progress during the period of October 1967 through March 1968 on a portion of a research program on combustion and combustion instability sponsored by the Office of Advanced Research and Technology of the National Aeronautics and Space Administration (NASA) under NASA Work Order 6030.

This report is released at the working level for information purposes and does not represent the final judgment of the Naval Weapons Center.

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1. INTRODUCTION

This report summarizes research conducted from October 1967 through March 1968 which was performed to assess the decomposition and deflagration behavior of ammonium perchlorate (AP). Crystalline AP, used in a granulated form, is the oxidizer and principal ingredient in most modern composite propellants and its behavior in the combustion zone greatly influences the overall combustion of the propellant. The results previously obtained may be found in Ref. 1 and 2.

This report may conveniently be divided into two parts, (1) low pressure decomposition studies and, (2) high pressure deflagration studies. The low pressure decomposition studies were concerned with (a) microscopic examination of the interface decomposition of pure single crystals¹ of AP in vacuum and (b) a scanning electron microscope (SEM) examination of the surface morphology of AP crystals subjected to high heating rates at atmospheric pressure. The high pressure deflagration studies included (a) the self-deflagration behavior of pure single crystals¹ of AP in a nitrogen atmosphere of $300 \leq p \leq 5000$ psia, and (b) the deflagration of AP crystals in methane atmospheres of $15 \leq p \leq 200$ psia.

2. AMMONIUM PERCHLORATE DECOMPOSITION

2.1. INTRODUCTION

The microscopic examination of the interface decomposition of AP single crystals was found to be an excellent method to measure the kinetic parameters of the decomposition process. Experiments have been made between 221°C and 231°C, temperatures below the phase transition temperature of 240°C, and at a pressure of one atmosphere (Ref. 1-2). During this reporting period the study of pure AP single crystal decomposition was continued in vacuum below the phase transition temperature. In addition, work on high temperature decomposition was initiated by heating single crystals with a flame or hot platinum. The topology of the decomposition was studied by SEM.

¹ The pure AP crystals used in the studies were grown by Dr. W. R. McBride of the Research Department, Michelson Laboratory, Naval Weapons Center, China Lake, California. A description of the technique is given in Ref. 1.

2.2. LOW TEMPERATURE DECOMPOSITION EXPERIMENTS²

During the decomposition of orthorhombic AP crystals at low vacuum (20 mm Hg) and at atmospheric pressure, a condensate was obtained on a cool quartz plate (Ref. 2) which was analyzed to be pure AP. This indicated an appreciable sublimation of AP even at atmospheric pressure and caused doubt about the role of sublimation in the measurement of decomposition rates. It is generally assumed (Ref. 3) that the first step of decomposition in the low temperature region is the dissociation of AP via proton transfer



followed by other reactions, such as desorption, condensed and/or gas phase reactions of the products. There are several methods which may be used to shift this equilibrium; for example, the addition of NH_3 to the gas phase (Ref. 4) or by changing the pressure of an inert atmosphere around the sample. In order to study the possible influence of pressure on the decomposition, the experiments were repeated at a vacuum pressure of 50×10^{-3} mm Hg and the results compared with the data obtained at atmospheric pressure.

The course of the decomposition at a pressure of 50×10^{-3} mm Hg was the same as under atmospheric pressure (Ref. 1). The sequence of events was:

1. Nucleation or initiation of decomposition sites on the different crystal faces.
2. Nuclei growth, disk-like on m-faces, streak-like on c-faces with the streaks running parallel to the b-axis.
3. Overlapping of the decomposition sites and development of a porous layer.
4. Interface decomposition between porous layer and undecomposed crystal cores.

However, there was a characteristic difference between decomposition at atmospheric pressure and under vacuum. At atmospheric pressure the porous residue layer grew linearly with time and at completion of the partial decomposition the porous residue occupied the volume of the original single crystal. Under a vacuum of 50×10^{-3} mm Hg the porous residue layer grew only to a thickness between 50 μ and 100 μ .

² This work was supported by Naval Ordnance Systems Command ORD-033 129/200 1/R001-06-01, Problem Assignment #5, and is reported here for continuity.

The interface decomposition rates in vacuum have been measured and compared with the values obtained at atmospheric pressure. Due to the vacuum sublimation of the residue layer the method of rate measurement was slightly modified. Instead of measuring the thickness of the porous layer as a function of time, the method used at atmospheric pressure, the decrease in size of the undecomposed core of an AP single crystal was determined as a function of time. This allowed the evaluation of the interface velocity since the original crystal size was known.

Figure 2.1a-d shows a crystal in an advanced decomposition state typical of partial decomposition in vacuum. The structure of the porous layer is not as smooth as under atmospheric pressure. One can clearly distinguish the m-faces with shallow dips from the c-faces with a streak structure. Figure 2.2 shows the penetration of the decomposition interface into AP single crystals as a function of time as measured at 221°C, 226°C, and 231°C and a pressure of 50×10^{-3} mm Hg. The penetration rates obtained from the slopes of the straight lines in Fig. 2.2 were entered into an Arrhenius diagram (see Fig. 2.3) from which the pre-exponential factor and activation energy for the interface decomposition under vacuum were calculated. In Table 2.1 the values are compared with those obtained and reported earlier (Ref. 1) for the decomposition at atmospheric pressure.

TABLE 2.1. Kinetic Parameters for the Decomposition of Pure AP Single Crystals at Atmospheric Pressure and 50×10^{-3} mm Hg

Pressure, mm Hg	Preexponential factor, cm/sec	Activation Energy, cal/mole
50×10^{-3}	2.9×10^3	21,100
≈ 730 (Ref. 1)	6.5×10^3	21,900

The activation energies are approximately the same and the preexponential factors differ only by a factor of two. This indicates that the same decomposition mechanism can be assumed in the pressure range investigated. Assuming dissociation by proton transfer as the decomposition mechanism, one can distinguish two systems (see Fig. 2.4). One system consists of three phases, namely AP single crystal, porous AP residue and a vapor phase and two components, namely NH_3 and HClO_4 . The other system consists of two phases (porous AP residue and vapor) and two components (NH_3 and HClO_4). The first system, in a simple model, would describe the interface decomposition whereas the second system would govern



(a)



(b)



(c)



(d)

FIG. 2.1. Ammonium Perchlorate Crystal in an Advanced State of Decomposition. Figures (a) shows the streak structures of a c-face and shallow dips on m-faces. Figures (b) through (d) are magnifications of the c-face in Fig. (a).

sublimation. Applying the phase rule, one can see that the interface decomposition has topochemical character. With three phases and two components the system has only one degree of freedom; there is only one dissociation pressure for a given temperature independent of the concentration of the solid phases (degree of decomposition). Likewise, the decomposition velocity of single crystalline AP and the formation velocity of porous residual AP have to be independent of the mass ratio of

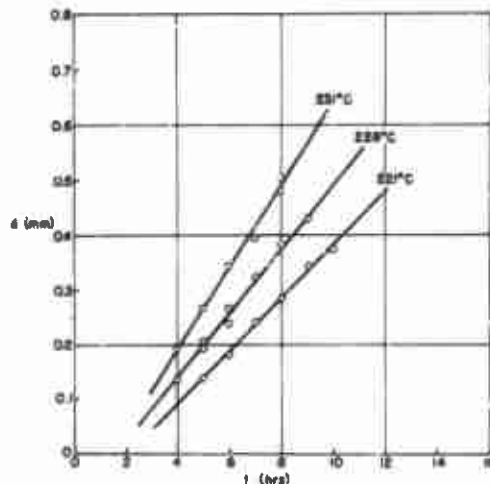


FIG. 2.2. Penetration of the Decomposition Interface into an Ammonium Perchlorate Single Crystal as a Function of Time. Vacuum decomposition at a pressure of 5×10^{-2} mm Hg. d = distance from crystal surface to interface.

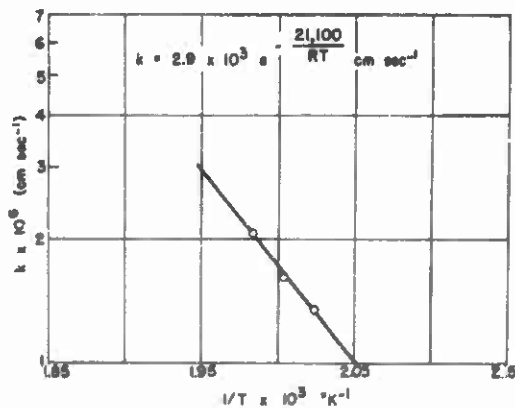


FIG. 2.3. Arrhenius Plot of the Rate of Decomposition of Orthorhombic Ammonium Perchlorate Single Crystals as a Function of Temperature. Vacuum decomposition at a pressure of 5×10^{-2} mm Hg.

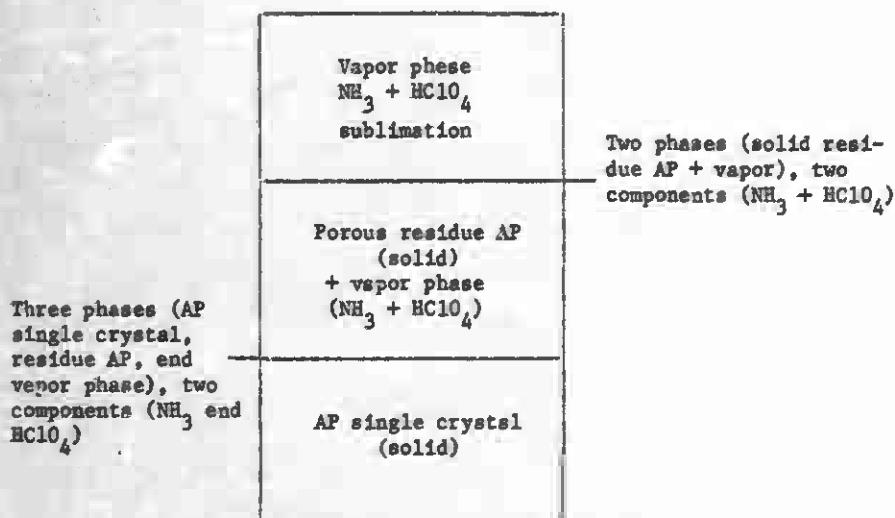


FIG. 2.4

the phases and both reactions have to occur at the same place. The only place possible is the contact surface between single crystalline AP and porous AP residue; in other words, the interface. This too would explain the pressure independence of the interface decomposition. According to this model, the developing AP residue would grow into the AP single crystal at the contact surface, thus consuming the single crystalline AP. That means the AP single crystal as a whole would be involved in the reaction. However, this has not been investigated yet. In addition, not much is known about the influence which AP decomposition products, other than NH_3 or HClO_4 , have on the decomposition reaction itself.

The experimental setup has been modified to allow the investigation of the interface decomposition of AP single crystals at elevated pressures up to 1000 psig. In addition an effort will be made to measure the thickness of the porous residue layer as a function of pressure below one atmosphere. Such information would differentiate between the contributions of sublimation and interface decomposition and possibly would permit the evaluation of the kinetic parameters of sublimation.

2.3. THE SURFACE STRUCTURE OF AP SINGLE CRYSTALS DECOMPOSED AT HIGH TEMPERATURES AND HIGH HEATING RATES

Whereas previous results were for the low temperature isothermal decomposition of AP single crystals, rocket propellants burn under very

high surface temperature gradients, high heating rates, and high pressure. In order to extend the study from isothermal experiments to high temperature, high heating rate decomposition, AP single crystals were heated by three different methods:

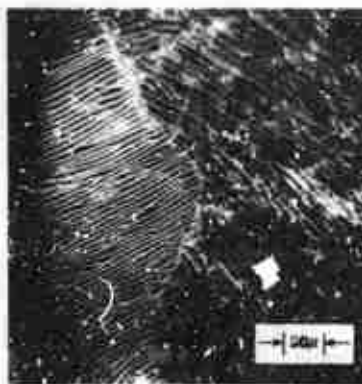
1. Directing a dry CO/O_2 flame against one of the crystal surfaces,
2. Dropping single crystals on a platinum surface preheated to approximately 700°C and,
3. Pressing a hot platinum wire ($\approx 700^\circ\text{C}$) into a single crystal.

The first method left the crystal surface open to the surrounding atmosphere so that the decomposition products could escape freely. In addition, steady state decomposition could be achieved. The second method of heating restricted the escape of decomposition products due to the hot platinum surface in contact with the crystal surface. This experiment could be compared with hot plate pyrolysis experiments done by other investigators. The skipping of the crystals on the hot platinum indicated the vigorous evolution of product gases (vapors) and caused a continuously changing heating rate. The third method produced the highest heating rates and restricted the escape of the reaction products to the greatest degree. In all three cases the crystals decomposed for a time and, after cooling, the surfaces were examined with the scanning electron microscope. As one can see in Fig. 2.5s-d through 2.12, the results, depending on the method of heating and decomposition, are quite varied.

On heating a crystal with a dry CO/O_2 -flame, the heated surface which was open to the atmosphere took on a glassy smooth appearance. In Fig. 2.5a-d part of an m-face is photographed at different magnifications. The great number of ridges which are roughly parallel within areas were assumed to be an artifact of cooling after heating was discontinued. It was assumed that they developed on the recrystallization of a liquid layer present during decomposition. However, direct microscopic observation of crystals decomposed with a flame showed that often the ridges were visible through most of the decomposition. Thus the ridges and domains cannot be assumed to be artifacts of the final cooling or of the phase transition from the cubic to the orthorhombic modification. Since the surface pattern was rapidly changing and details could not be resolved by visual observation, a movie camera will be combined with the microscope to overcome the problem. Although there was strong indication of a liquid layer on the surface of a crystal decomposed by a hot flame, final proof has still to be established in this case. Figure 2.6a-d shows a corner section of the same m-face. Here again, the more or less parallel ridges (not as long and sharp as near the center of the m-face) contain transverse ripples with pits which look like collapsed parts of the surface. From the rupture line the thickness of the layer was estimated to be approximately $1/2 \mu$. Figure 2.7a-d shows a c-face of an AP crystal heated with the same CO/O_2 flame.



(a)



(b)



(c)



(d)

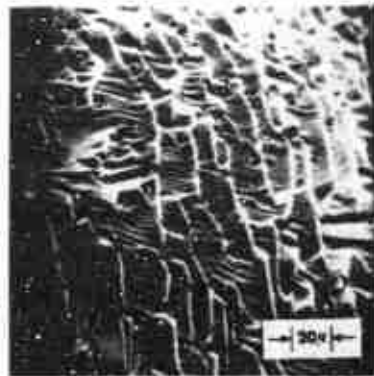
FIG. 2.5. m-Face of AP Single Crystal After Heating With a CO/O_2 -Flame.

Beside smooth areas and parallel ridges, one can see rhombohedral structures typical for c-faces. One of the rhombohedral pits is shown in magnification. However, the porous layer structure, which is so characteristic for the low temperature decomposition, was not observed on either m- or c-faces.

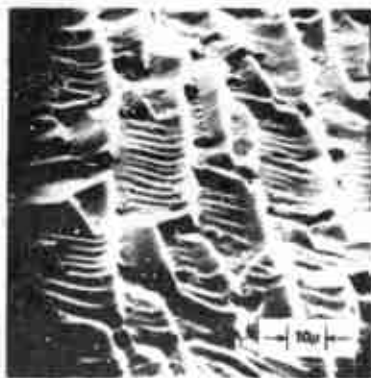
There is a major change in appearance if the surface is not heated openly but by contact with a hot plate. Photographs of crystals heated by the hot platinum plate demonstrate this change. Figure 2.8a-d shows



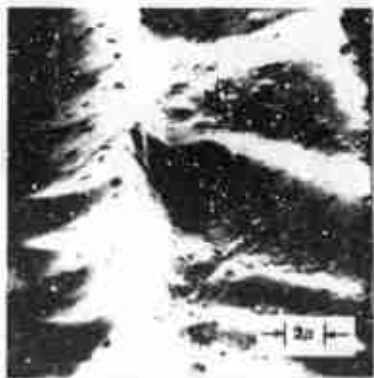
(a)



(b)



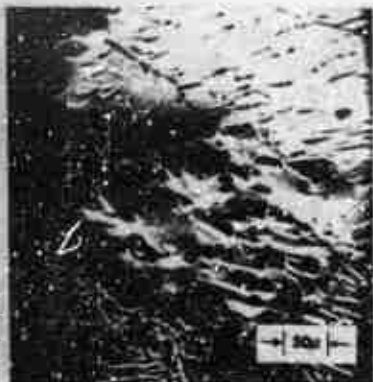
(c)



(d)

FIG. 2.6. Corner Section of m-Face of AP Single Crystal After Heating With a CO/O₂-Flame (Same m-Face as in Fig. 2.5).

part of an m-face at different magnifications. Characteristic features are the dome-shaped structures which cannot be explained without assuming some kind of highly viscous liquid. In addition, one can see porous areas of the type observed for low temperature decomposition ($T < 240^{\circ}\text{C}$). Furthermore, the porous parts of the m-face seem to occur in the depressed areas whereas the half-domes constitute the elevated areas. This can be seen more clearly in Fig. 2.9a-d which shows a corner section of the same m-face. The half-spheres, due to the good contact with the hot metal, have to be considered as hot spots which emitted vapors thus causing the



(a)



(b)



(c)



(d)

FIG. 2.7. c-Face of AP Single Crystal After Heating With a CO/O₂-Flame.

crystal to skip. The depressions would be at a lower temperature leading to a porous surface, as observed for the low temperature decomposition. Figure 2.10a-d shows a c-face heated by the same method. The features are very similar to those observed on m-faces. In addition, decomposition streaks parallel to the b-axis of AP can be seen which are characteristic for the c-face. This indicates low temperature decomposition occurred in this part of the crystal and hence large temperature gradients existed across the surfaces.



(a)



(b)



(c)



(d)

FIG. 2.8. m-Face of AP Single Crystal After Heating With a Hot Plate.

Finally, heating by pressing a hot wire into the crystal greatly restricted the escape of products between wire surface and crystal and, at the same time, there was an open surface at the top of the crystal. The volume of crystal decomposed was approximately the volume displaced by the wire in the crystal. The products escaped through the gap between wire and crystal towards the open atmosphere where expansion, rapid cooling and condensation occurred. A picture of the resultant AP surface is shown in Fig. 2.11a-d. At low magnification a hole can be seen on the m-face of a crystal surrounded by the deposit of decomposition products.

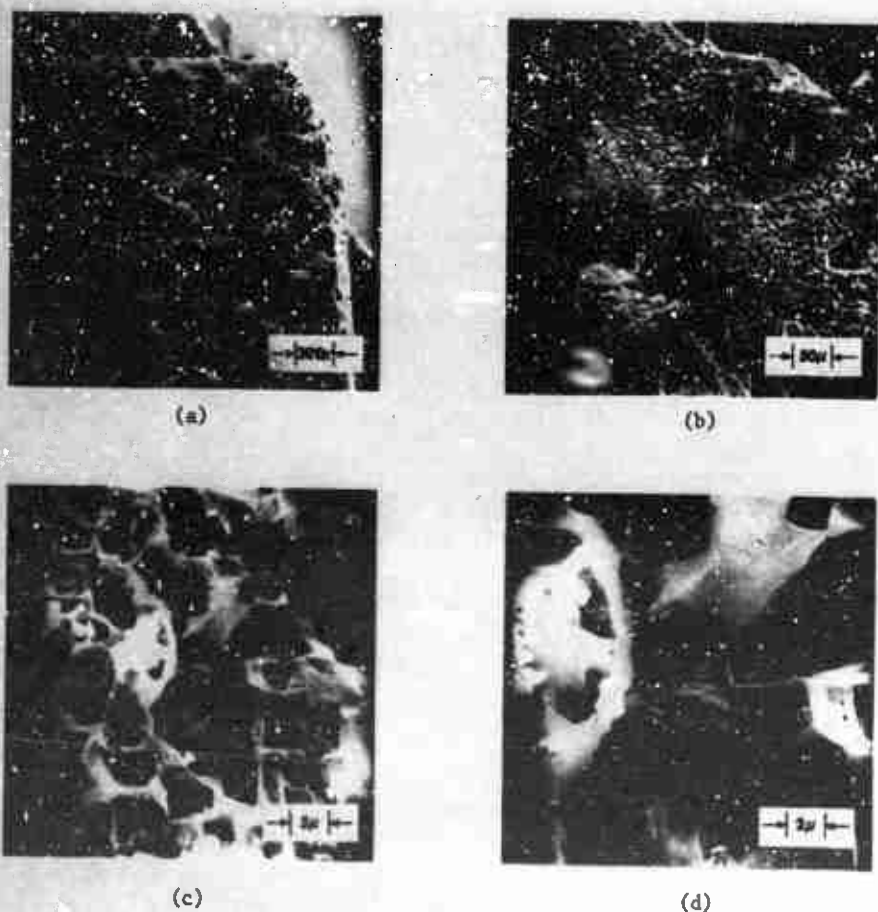
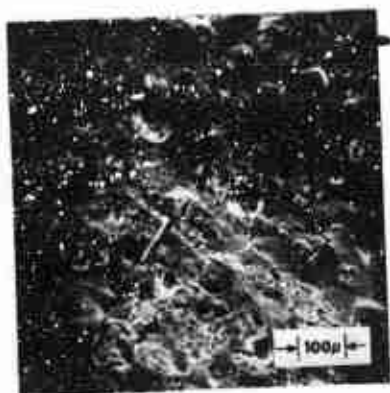


FIG. 2.9. Corner Section of m-Face of AP Single Crystal After Heating With a Hot Plate (Same m-Face as in Fig. 2.8).

Especially interesting is a section of the rim of the hole in the center of the upper left picture. From the structure, its development can be inferred as a pattern one obtains if a highly viscous liquid is extruded through an annulus. Immediately after leaving the hole the liquid crystallized by very rapid cooling to a highly porous material (Fig. 2.11).

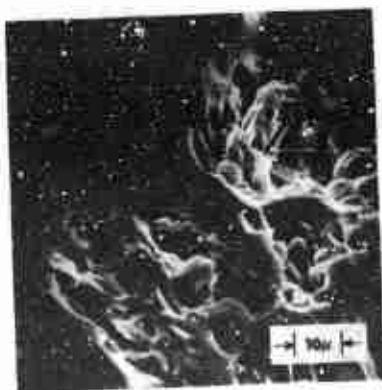
The structure is identical to a crystal partially decomposed at a low temperature (Ref. 1). Figure 2.12a-d shows another section around the same hole. Again the spherical shape of the deposit implies that



(a)



(b)



(c)



(d)

FIG. 2.10. c-Face of AP Single Crystal After Heating With a Hot Plate.

a liquid phase was present. Although a deposit obtained by crystallization of a condensate from the vapor phase shows a porosity (Fig. 2.13a-d) similar to the one in Fig. 2.12, it does not have a comparable macroscopic structure (e.g., big porous spheres).

A few examples of similarities between the decomposition, as manifested in examples described above and the high pressure self-sustained deflagration of AP crystals, have been found. However, as yet, the number of examples is not sufficient to establish correlations between decomposition and self-sustained deflagration.



(a)



(b)



(c)



(d)

FIG. 2.11. m-Face of AP Single Crystal After Heating With a Hot Wire.

2.4. SUMMARY

The investigation of the low temperature decomposition showed that the interface decomposition which resulted in a porous residue is pressure independent at and below one atm pressure whereas sublimation of the residue containing approximately 65% of the original AP weight is highly pressure sensitive. Assuming an identical first reaction step in both cases, that is dissociation of AP by proton transfer, the kinetic parameters can be ascribed to this step. By applying high heating rates and



(a)



(b)



(c)



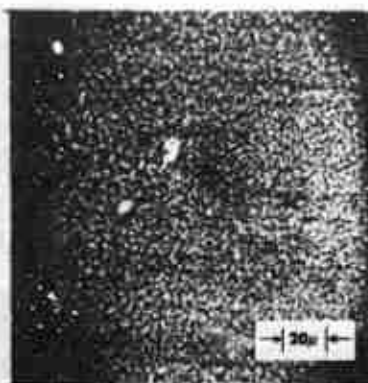
(d)

FIG. 2.12. m-Face of AP Single Crystal After Heating With a Hot Wire (Section of Fig. 2.11a).

temperatures it was found that a liquid phase is involved in this kind of decomposition even at atmospheric pressure. Although there are similarities to the phenomenology of high pressure self-sustained deflagration of AP single crystals, it is too early to draw definite conclusions.



(a)



(b)



(c)



(d)

FIG. 2.13. Ammonium Perchlorate Condensed From the Vapor Phase.

3. AMMONIUM PERCHLORATE DEFLAGRATION

3.1. INTRODUCTION

Previous investigations (Ref. 1, 2, 5, and 6) provided evidence that a thin layer of liquid material was present on the surface of pure AP crystals undergoing self-deflagration. Whereas no such melt has been reported for isothermal decomposition studies, it has been observed when the AP was rapidly heated. In the case of self-deflagration, gaseous decomposition products were trapped within this liquid resulting in a visible froth. It has been proposed that this froth may contribute significantly to the heat transfer necessary to sustain deflagration (Ref. 2). The investigations conducted during this reporting period were designed to provide information concerning the (1) deflagration rate of AP as a function of pressure, (2) energy feedback mechanism prevalent at different pressures, (3) mechanisms responsible for the low pressure deflagration limit and (4) deflagration behavior of AP in methane atmospheres at pressures below the low pressure self-deflagration limit.

3.2. DEFLAGRATION RATE OF AMMONIUM PERCHLORATE

In this investigation single crystals of AP were deflagrated in the apparatus described in Ref. 1 and deflagration rates were obtained from high speed motion pictures. Advantages of the technique of high speed motion picture photography over other methods of burning rate measurement include (1) the measurement of surface regression to within ± 100 microns, (2) elimination of discontinuities such as fuse wires or thermocouples, and (3) a visual record of the test which permits rejection of the run if the sample departed from one-dimensional regression (coning or cupping of the surface), or if anomalous nonsteady state phenomena (stop and go burning) occurred. The movies also allowed observation of the surface during deflagration of the sample.

The burning rate versus pressure results of this investigation, together with the results of other investigations, are presented in Fig. 3.1. The rate results obtained in this study agree with those obtained by others in the regime $275 < p < 2000$ psia.

Above 2000 psia the data obtained using uninhibited single crystals of pure AP agree qualitatively with the data of Bobolev, et al (Ref. 7), Glaskova (Ref. 8), and Friedman, et al (Ref. 9), all of whom used pressed pellets. The data obtained by Levy and Friedman (Ref. 10) using

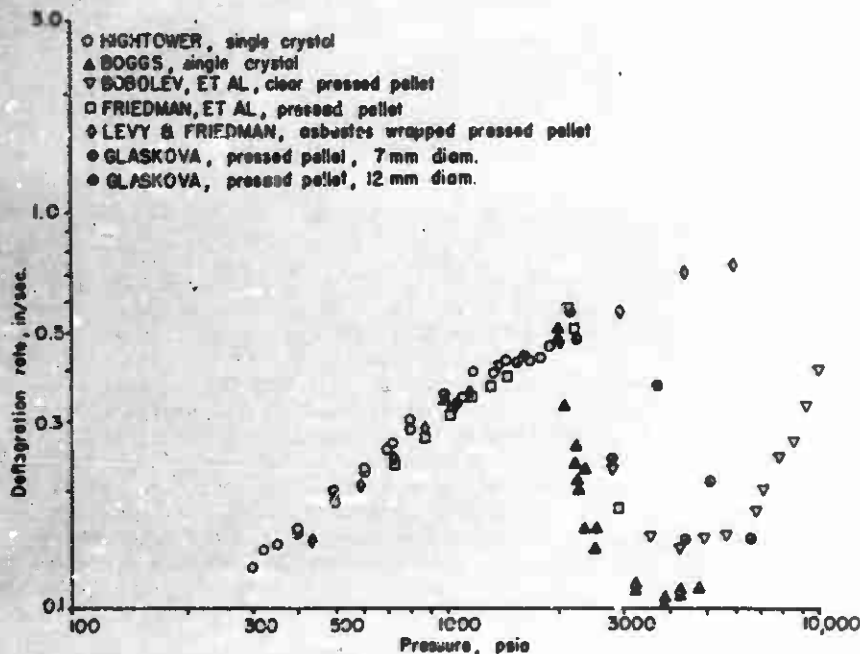


FIG. 3.1. Deflagration Rate of Ammonium Perchlorate Deflagrated in a Nitrogeo Atmosphere.

asbestos wrapped pressed pellets deviate significantly from that of the other investigators. Because of Friedman's (Ref. 9) low data point (~ 0.18 in/sec at 2800 psia (Fig. 3.1)) and the extinguishment behavior they observed, they concluded that convective cooling along the sides of the sample was responsible for the low point and extinguishment and that such cooling could be eliminated by the asbestos wrapping.

Glaskova (Ref. 8), in an investigation of the effects of sample size and wrapping on the deflagration rate, concluded that below 500 atm (approximately 7500 psi) the loss of heat by the sample to the surroundings played an insignificant role on the combustion rate of AP. Glaskova's tests to determine the effect of sample wrapping produced the data presented in Fig. 3.2. The curves exhibit a negative slope for pressures above 2000 psia, except for the fluorinated lubricant, even though heat was added at the sample boundary by the combustion of the Plexiglas and organic varnish shells. Glaskova asserted that the difference in the amount of heat liberated during the combustion of the various shells and of the fluorinated lubricant accounted for the difference in rates.

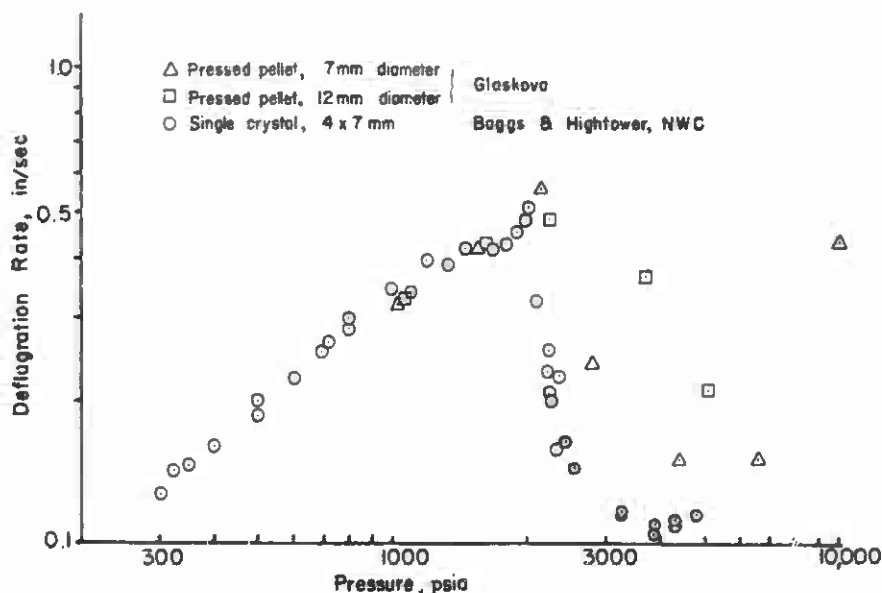


FIG. 3.2. The Effect of Sample Size on the Deflagration Rate of Ammonium Perchlorate.

The movies obtained in this investigation verify Glaskova's conclusions: no pronounced cupping of the crystal surface (which would occur if convective cooling were of consequence) was observed. Thus the assertion that the negative slope of the deflagration rate curve is due to convective cooling along the sides of the sample has not been substantiated.

3.3. MECHANISMS WHICH DETERMINE THE DEFLAGRATION RATE

It has been hypothesized (Ref. 1) that energy transfer to the self-deflagrating AP crystal occurs primarily in two ways: (1) by conductive heat transfer from the gas phase to the condensed phase and (2) by exothermic gas phase reactions which occur in what might be called a pseudo-condensed phase. This latter phase consists of a liquid layer with entrapped gas bubbles whose nucleation and growth within the melt produce a froth. Whereas one can readily estimate the amount of energy conducted from a reaction zone (flame), which is a given distance from the surface, through the gas phase to the crystal (Ref. 12), one has difficulty in assessing the amount and manner of energy transfer taking place in this froth. Before any type of thought as to the magnitude and manner of energy transfer within the froth can be initiated, characteristics of

this froth must be known. High speed motion pictures were taken of the deflagrating crystals in order to study the dynamic behavior of the surface and thermally quenched samples were examined using a SEM in order to determine the microscopic surface details associated with the combustion. The window bomb described in Ref. 1 served as the combustion chamber, and the cinephotography was performed with magnifications of 2X and 4X and framing rates up to 4000 frames/sec. The specimens examined using the SEM were obtained by thermally quenching a self-deflagrating crystal in a copper vise (Fig. 3.3); the quench occurred 1-2 mm above the vise. Such a quenching technique overcomes many of the drawbacks associated with the quenching by rapid depressurization previously used.

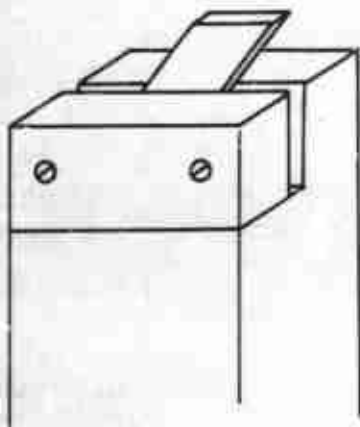


FIG. 3.3. Copper Vise Used to Thermally Quench Ammonium Perchlorate Samples.

In order to facilitate discussion of the observations and the energy transfer inferred from these observations, it is convenient to divide the deflagration rate curve into four regimes (Fig. 3.4): (I) constant positive slope of $\ln r$ versus $\ln p$ curve in the region $300 < p < 900$ psia, (II) decreasing positive slope of the curve with increasing pressure for the range $1000 < p < 2000$ psia, (III) negative slope for $2000 < p < 4000$ psia, and (IV) increasing positive slope for $p > 4000$ psia.

3.4. REGIME I ($300 < p < 900$ PSIA)

The quenched surface of a single pure AP crystal burned in a 314 psia nitrogen atmosphere is shown in Fig. 3.5a-f. Figure 3.5a shows the overall detail of the sample. It should be noted that the edges of the sample

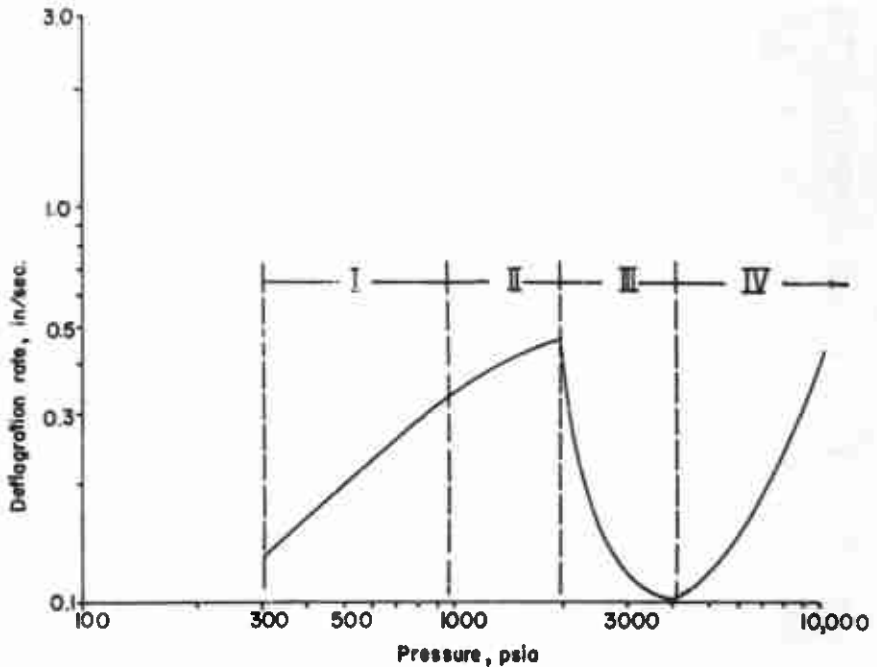


FIG. 3.4. The Four Regimes of the Deflagration Rate of AP Deflagrated in a Nitrogen Atmosphere.

(in contact with the copper vise) experienced a more rapid quench than did the interior. This is due to the contact of the edges of the samples with the jaws of the vise. The bubbles clearly indicate that gas was evolved through a molten layer. Whereas that portion of the sample which was first cooled exhibits bubbles and froth, the interior portion which was more slowly quenched (Fig. 3.5c and 3.5f) exhibits the structure one might expect if the liquid surface were slowly cooled to form a crust while at the same time gas was still emanating from below this cooled layer. The holes seen in Fig. 3.5c can also be seen within the domed structure in Fig. 3.5f and it is thought that these holes extend down to what had been the solid-liquid interface.

A sample quenched at 600 psia (Fig. 3.6a-f) is seen to possess structure similar to that of the 314 psia sample. Once again bubbles were formed at that portion of the sample which was most rapidly quenched and a rounded, crusted structure resulted on the more slowly quenched interior. Again gas liberation sites may also be seen.

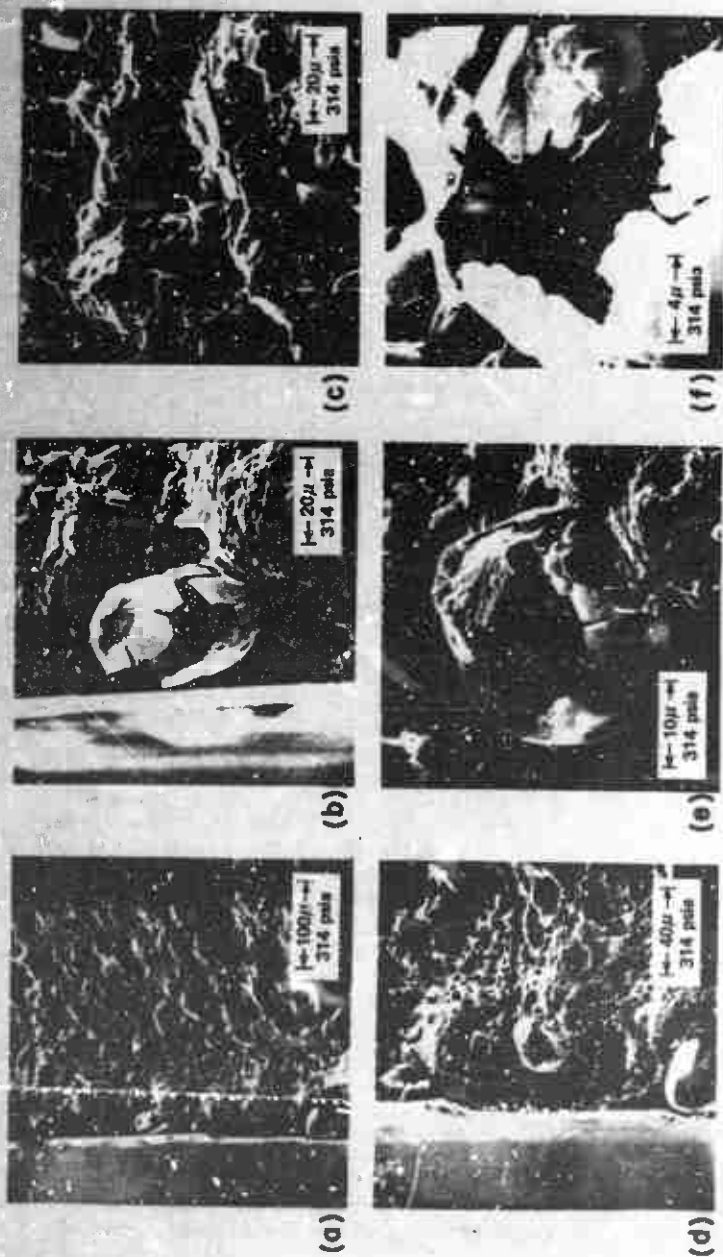


FIG. 3.5. Surface Morphology of Ammonium Perchlorate Samples Which Were Thermally Quenched While Self-deflagrating in a 314 psia Nitrogen Atmosphere.

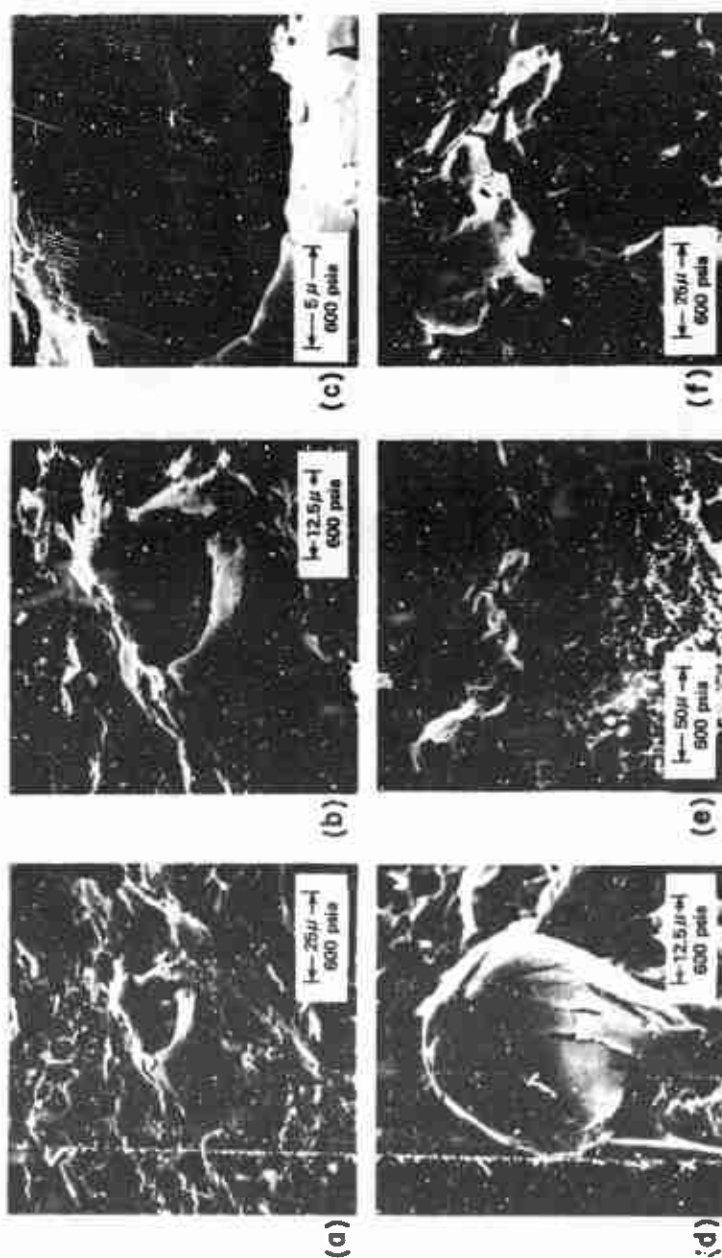


FIG. 3.6. Surface Morphology of Ammonium Perchlorate Samples Which Were Thermally Quenched While Self-deflagrating in a 600 psia Nitrogen Atmosphere.

In this regime ($300 < p < 900$ psia) characterized by $r = ep^n$ it appears that energy release takes place in the pseudo-condensed phase and adds to any contribution from the gas phase. The SEM pictures indicate that at these lower pressures a gas-entrapping melt was present as evidenced by the bubbles frothy appearance (Fig. 3.5-3.6). Because these samples were obtained from a thermal quench rather than depressurization quench, the bubbles are not an artifact of the method of quench, as might be the case of quench by depressurizing the test chamber. Perhaps of more significance than the bubbles, are the gas liberation sites (holes) apparent at the bottom of the bubbles (Fig. 3.5f and 3.6c). The depth of these sites suggest that the gas which was entrapped within the liquid had its origin at the solid surface and progressed through the liquid. Although it is not readily apparent when these few SEM pictures are viewed, the other pictures obtained during this investigation indicated that the thickness of the liquid layer decreased with pressure. The sample pictured in Fig. 3.7s-c was quenched at 900 psi and represents a transition between the samples of Regime I and Regime II. The SEM photographs indicate that spots of minimum regression may have existed on the surface but the motion pictures indicate that a melt covered the entire surface at this pressure. By combining the seemingly disparate results, the following interpretation for the behavior of the self-deflagrating crystal at this transition pressure is proposed. Consistent with the observation that the melt thickness decreases with pressure increase, it seems reasonable to assume (especially when the samples of Regime II are viewed) that at this pressure the melt has become extremely thin. Due to the thinness of the layer, cool spots may have developed during quench and the melt adjacent to these areas was drawn to and solidified at the cool spots. Other samples will be obtained at 800 psi and 1000 psi in order to assess the extent of this transitional behavior.

3.5. REGIME II ($1000 < p < 2000$ PSIA)

Whereas the surface observed in Regime I consisted of a froth that covered the entire surface, the surface of Regime II samples was characterized by an unchanging pattern of ridges and valleys. The SEM photographs taken of the samples quenched in this regime present a continuous development of surface pattern with pressure as illustrated in Fig. 3.8s-c. All of the samples had ridges and sites of activity in the valleys, although at the higher pressure the ridges appear to be almost vestigial. It is important to note that the reaction seems to take place over part of the surface only and the areas of liquid have a higher regression than do the ridges. Also of note is the difference of character of the ridges at these various pressures. The length of each ridge seems to be an inverse function of pressure; the ridges at 1200 psia are well over 200 μ in length whereas at 1800 psia very few are longer than 100 μ . The elevation of the ridges over the valleys also seems to be inversely proportional to pressure. Just as the characteristics of the ridges are a function of pressure, the activity sites within the valleys are also

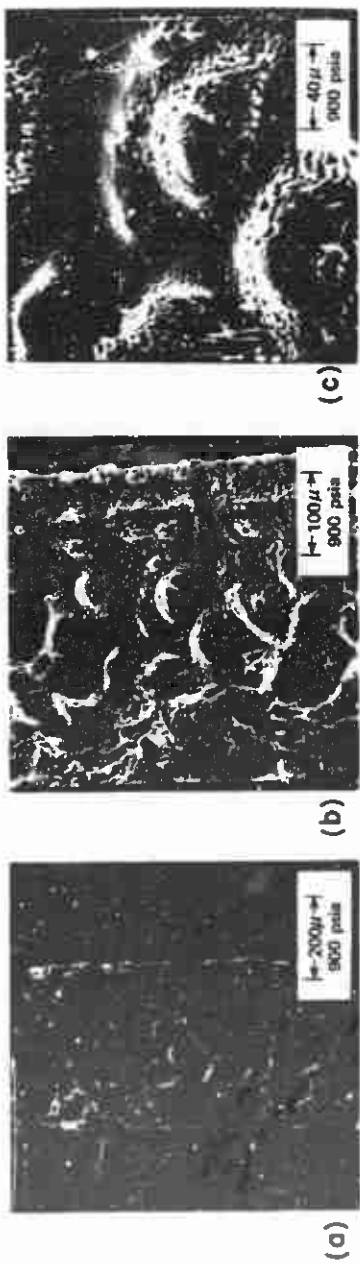


FIG. 3.7. Surface Morphology of Ammonium Perchlorate Samples Which were Thermally Quenched While Self-deflagrating in a 900 psia Nitrogen Atmosphere.



FIG. 3.8. Surface of Regime II Samples Which were Thermally Quenched While Undergoing Self-deflagration. Note the ridges and activity sites.

pressure dependent. The samples quenched at 1200 psie (Fig. 3.9a-f) show a frothy material in the valley similar to the surface of samples from Regime I. The samples quenched at 1500 psie (Fig. 3.10a-f), while having the gas liberation sites surrounded by the frothy material similar to the 1200 psie samples, also show activity sites which contain depressions of a rather shallow nature and holes that are filled with pillars. The holes seen in the valleys of the 1800 psi sample (Fig. 3.11a-f) are of the same size as those seen on the 1200 and 1500 psi samples, and they have the structure obtained at high heating rates where the reaction causes the vigorous expulsion of melt by gas in a manner analogous to the expulsion of lava by a volcano. Also in evidence are the pillars which will be seen to characterize the samples quenched at higher pressures.

Since the maximum regression takes place in the valleys, it would appear that the ridges are cooler than the valleys. Consistent with the idea that the ridges are "cool", the motion pictures taken at 4X magnification indicate that often these ridges did not burn at all, rather they peeled off from the surface unburned. The motion pictures also indicated that the pattern of ridges and valleys was spatially invariant with time. Consistent with the observations made for Regime I that the liquid seems to decrease with increasing pressure, it was hypothesized that at some critical value of liquid layer thickness the cohesive forces would be greater than the adhesive forces and the liquid film would be drawn up into droplets. If maximum energy feedback, hence maximum regression rate, occurred through the liquid then this would explain the "fingerprint" surface pattern of ridges and valleys observed by Hightower (Ref. 13). The unchanging pattern of ridges and valleys and the peeling off of unburned ridges from the surface lend credence to this preliminary hypothesis. Further investigation test and expand this hypothesis is currently being pursued.

3.6. REGIME III ($2000 < p < 4000$ PSIA)

As evidenced by Fig. 3.1, this is the region of greatest uncertainty as to the magnitude and slope of the deflagration rate curve. The difficulty in igniting and sustaining a burn in this region encountered by Friedman, et al, (Ref. 7) when they used 4 mm diameter pressed pellets was largely overcome by using samples of 4 mm x 4 mm or larger cross-section. The results of this investigation partially explain this behavior and future effort will be devoted to gaining further knowledge of the responsible combustion mechanisms.

Whereas the macroscopic regression in Regimes I and II was steady and propagated at a uniform rate over the surface, high speed motion pictures taken of samples deflagrating at the pressures of this regime displayed a local regression which took place at separate concave sites. The pulsation, which was also noted by Bobolev, et al (Ref 7), and Glaskova (Ref. 8), was due to the reaction taking place at certain sites, ceasing at those

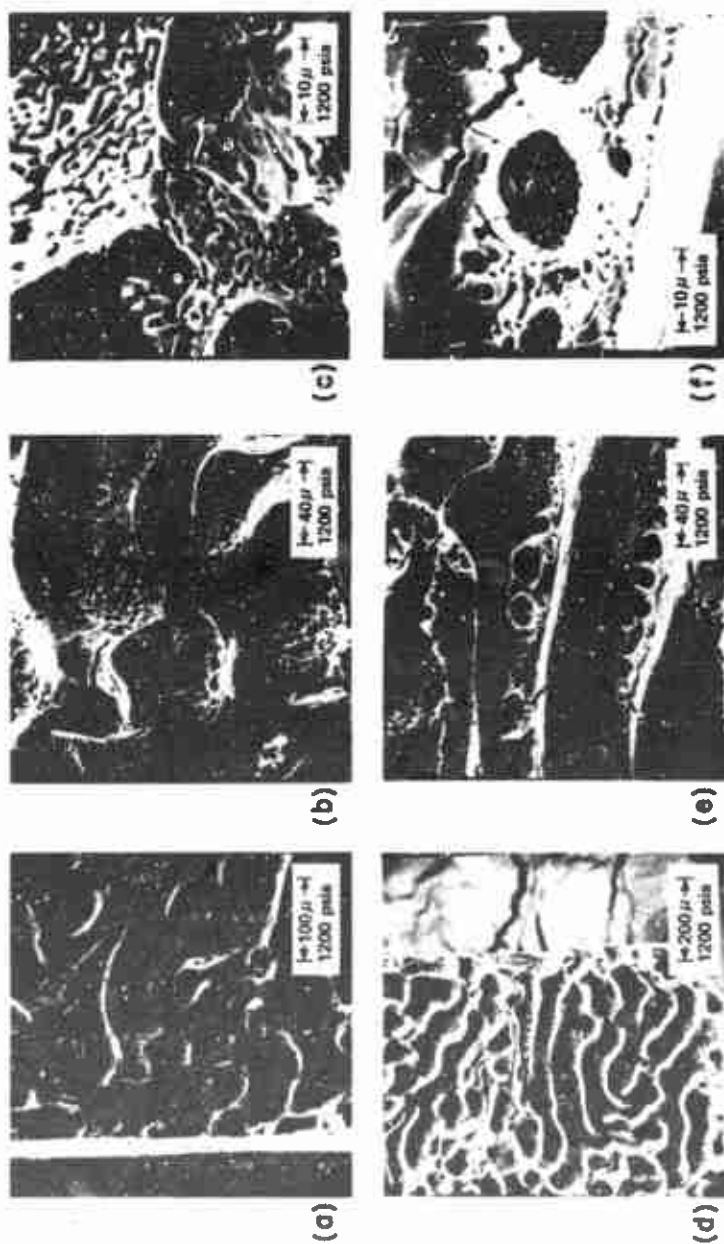


FIG. 3.9. Surface Morphology of Ammonium Perchlorate Samples Which Were Thermally Quenched While Self-deflagrating in a 1200 psia Nitrogen Atmosphere.

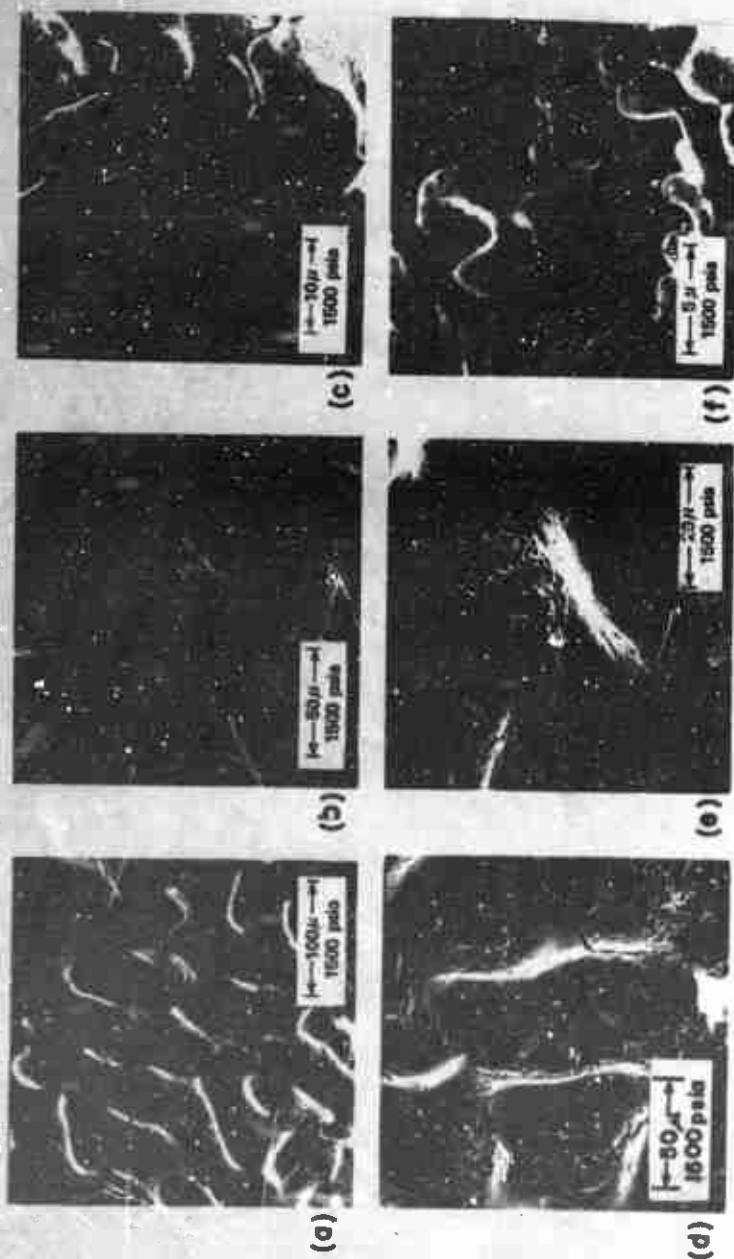


FIG. 3.10. Surface Morphology of Ammonium Perchlorate Samples Which Were Thermally Quenched While Self-deflagrating in a 1500 psia Nitrogen Atmosphere.

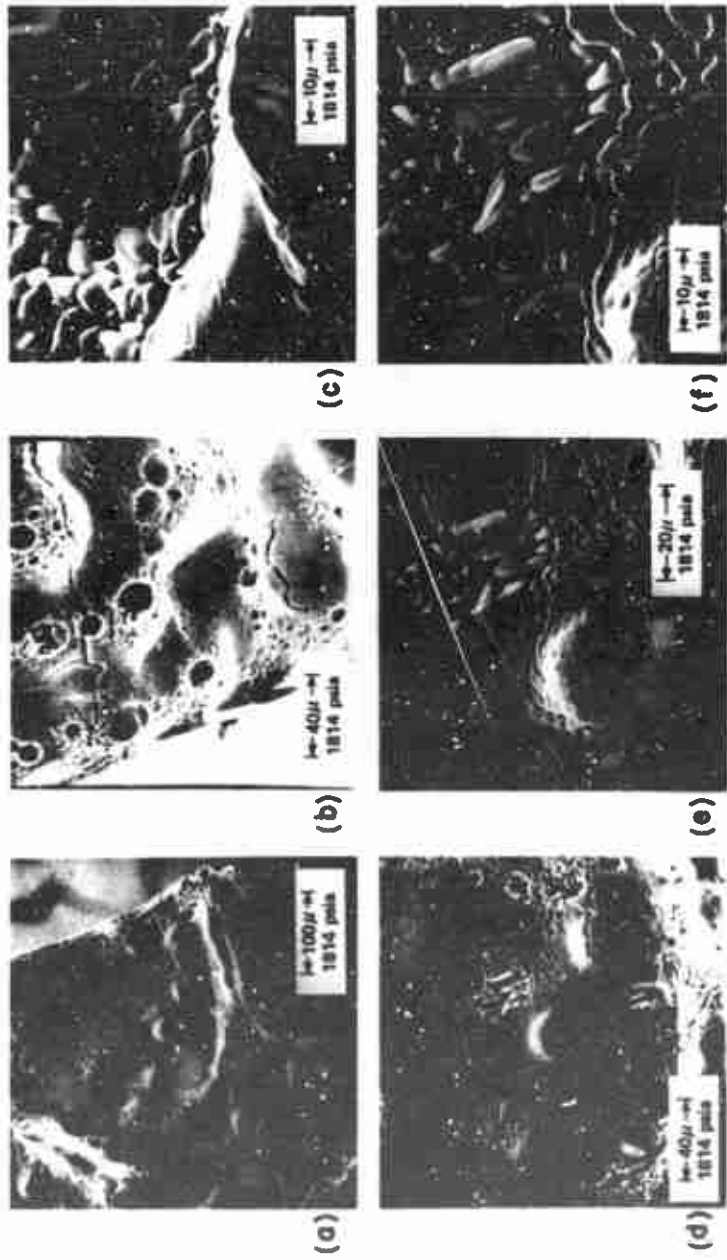


FIG. 3.11. Surface Morphology of Ammonium Perchlorate Samples Which Were Thermally Quenched While Self-deflagrating in a 1800 psi Nitrogen Atmosphere.

sites and taking place at other sites on the surface. Movies taken without external illumination (self-illumination) of the self-deflagrating crystals showed an orange flame which changed location on the regressing surface with time.

Scanning electron microscope pictures of the sample obtained at 2200 psi (Fig. 3.12a-f) illustrate three basic types of structures: (1) holes similar to those apparent on the SEM photographs for 1800 psi (Fig. 3.12b and 3.12c), (2) pockets of randomly oriented pillars (Fig. 3.12e and 3.12f), and (3) small peaks (Fig. 3.12a and 3.12d). The pillars within the pockets of maximum regression are associated with a flame existing close to the surface as shown in the following figures. The peaks are similar to the vestigial ridges seen on the 1800 psi sample.

The surface of the sample which was thermally quenched at 2800 psig is presented in Fig. 3.13a-f and 3.14a-f. In Fig. 3.13b the sample is graphically depicted and the leaders to the other pictures indicate which portion of the surface was being examined. The area of maximum regression, that portion where the flame was observed, possesses the pillar structure of Fig. 3.13c and 3.13f. The area immediately adjacent to the region of maximum regression, Fig. 3.13e, indicates that a melt may have been present in this area. Figure 3.14 presents enlarged views of the pictures of Fig. 3.13.

The sample which was quenched at 3300 psig is presented in Fig. 3.15a-f, 3.16a-f, and 3.17a-f. Once again an area of maximum regression (Fig. 3.15a and 3.15b) which possesses the needle structure (Fig. 3.15d, 3.15e, and 3.15f) may be seen. Figure 3.15d shows the structure found at the bottom of the pit whereas the structure found on the side of the depression is shown in Fig. 3.15f. It should be noted that the "lip" seen in Fig. 3.15b fortuitously provides a chance to look at Fig. 3.15c which was partly accessible to the flame while a portion was shielded from the flame. That portion which was open to the flame possesses the needle structure previously seen while the shielded portion has a different appearance. Other portions of the surface are seen in Fig. 3.16. The structure of the material exposed to the flame (Fig. 3.16d, 3.16f, and 3.17a, b, d, and e) differs from the material far from the flame (Fig. 3.16a, 3.17c, and 3.17f).

The cinephotography indicated that the pulsating surface regression was caused by a flame which changed location on the surface. The movies also show that this flame is always associated with the maximum regression, i.e., it existed in the pits. When subjected to SEM examination, the area of maximum regression (associated with the flame) exhibited a microstructure of needles while the area immediately adjacent possessed a structure which indicated that a melt may have been present. These observations made it possible to formulate a phenomenological explanation for the deflagration in this regime. To facilitate discussion a time sequence of the typical profiles of the crystal (obtained directly from the high speed movies) with flame sites sketched in is shown in Fig. 3.18.

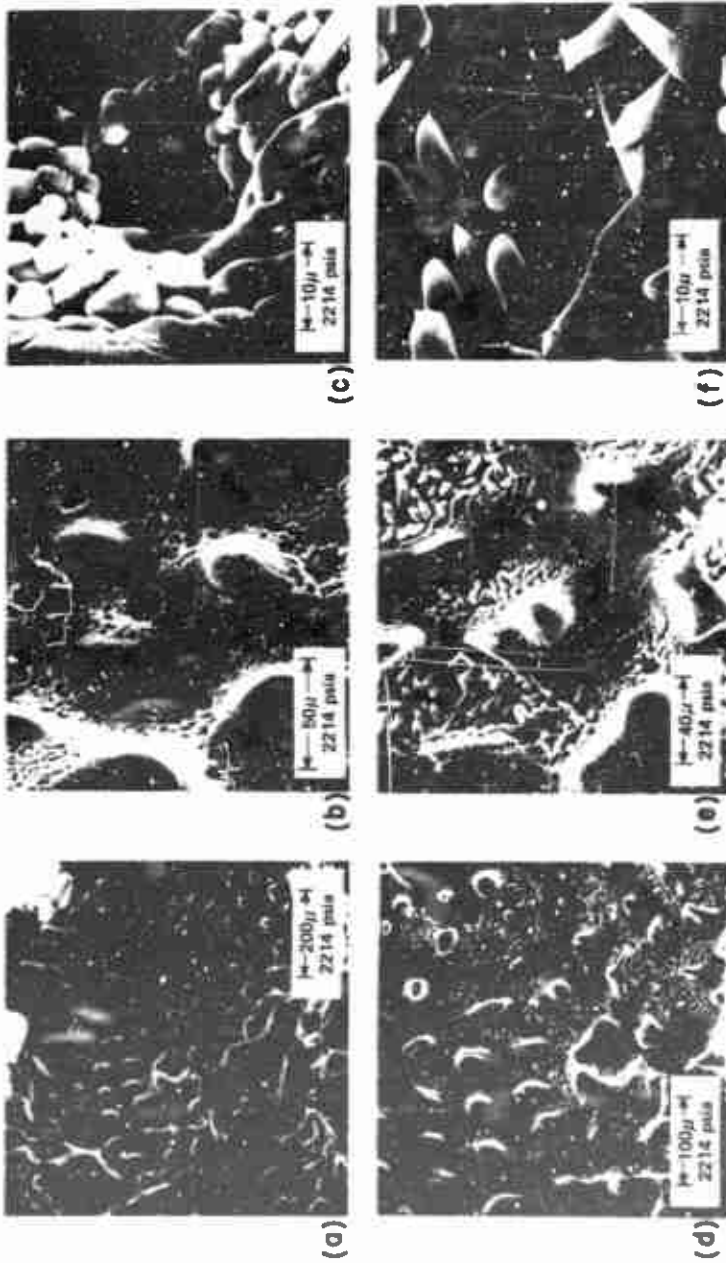


FIG. 3.12. Surface Morphology of Ammonium Perchlorate Samples Which Were Thermally Quenched While Self-deflagrating in a 2200 psi Nitrogen Atmosphere.

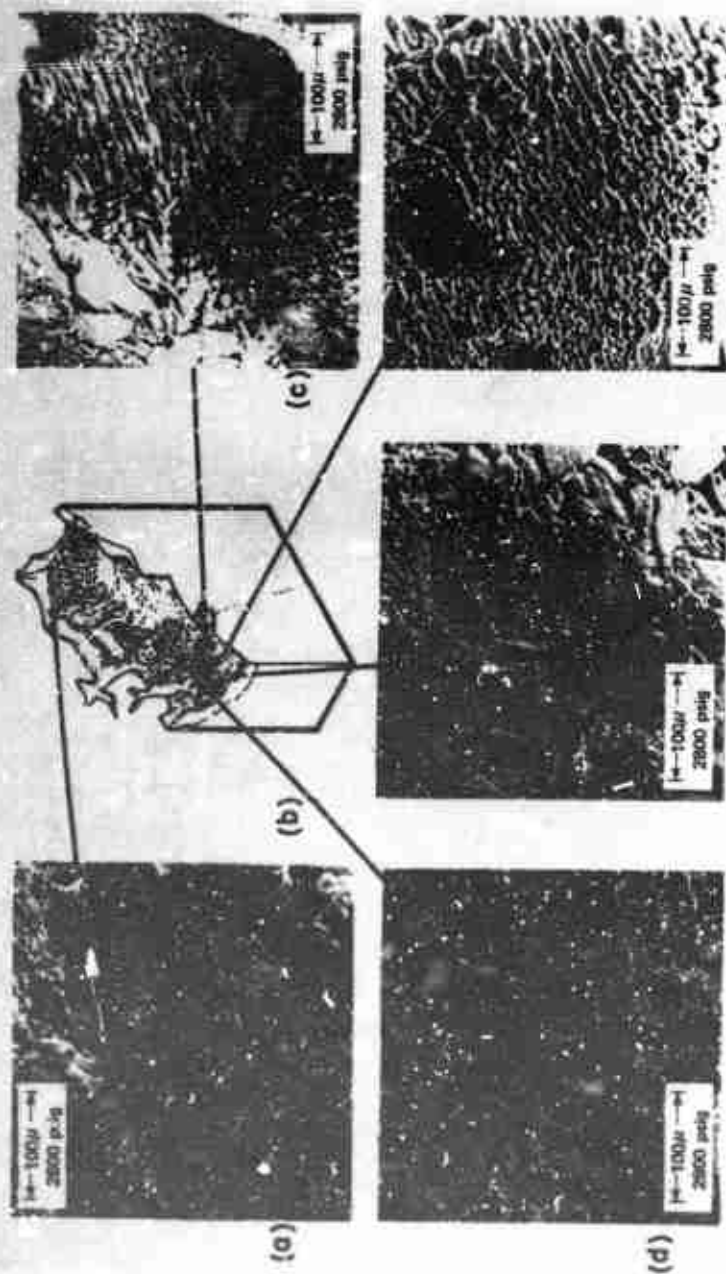


FIG. 3.13. Surface Morphology of Ammonium Perchlorate Samples Which Were Thermally Quenched While Self-deflagrating in a 2800 psia Nitrogen Atmosphere.

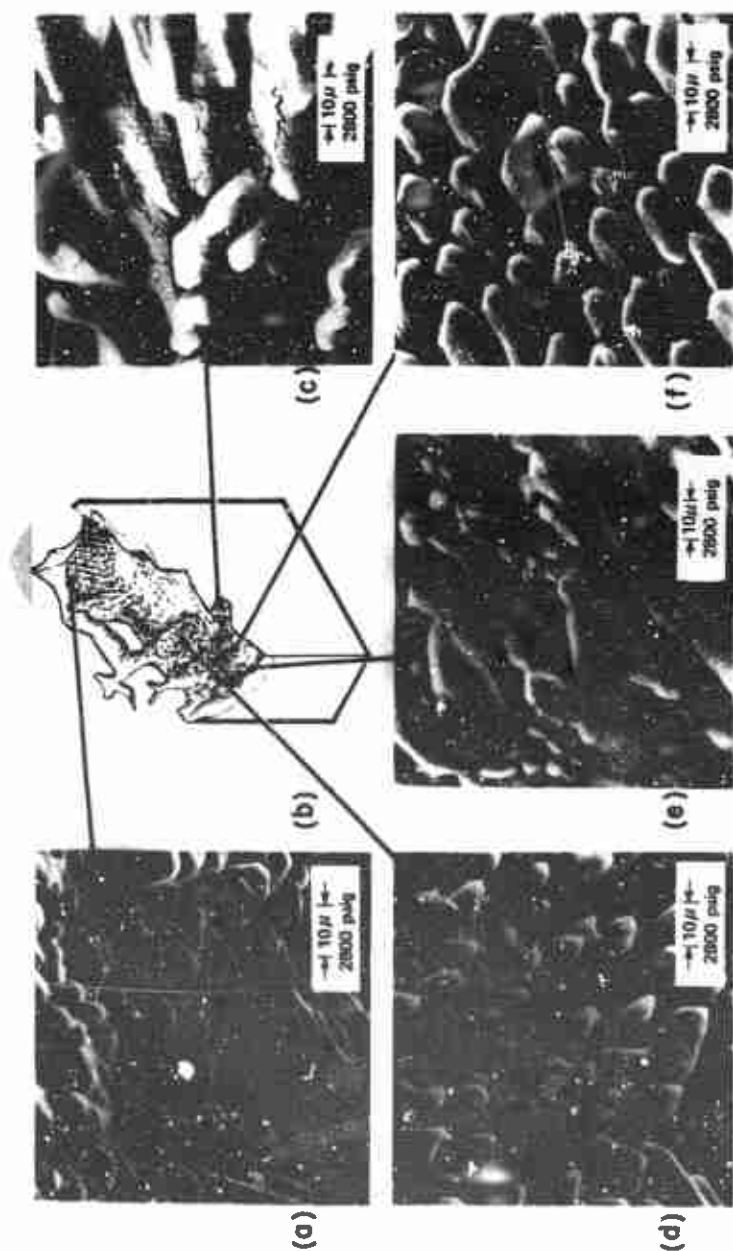


FIG. 3.14. Surface Morphology of Ammonium Perchlorate Samples Which Were Thermally Quenched While Self-deflagrating in a 2800 psia Nitrogen Atmosphere.

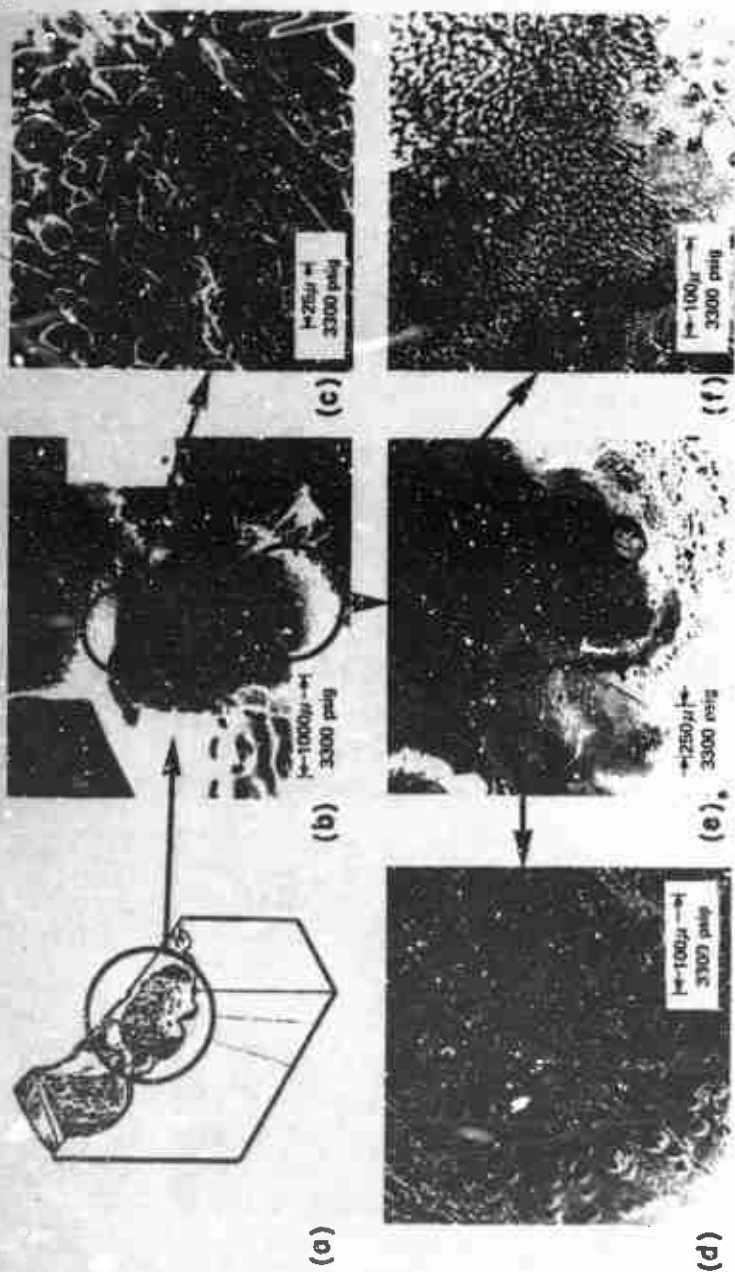


FIG. 3.15. Surface Morphology of Ammonium Perchlorate Samples Which Were Thermally Quenched While Self-deflagrating in a 3300 psig Nitrogen Atmosphere.

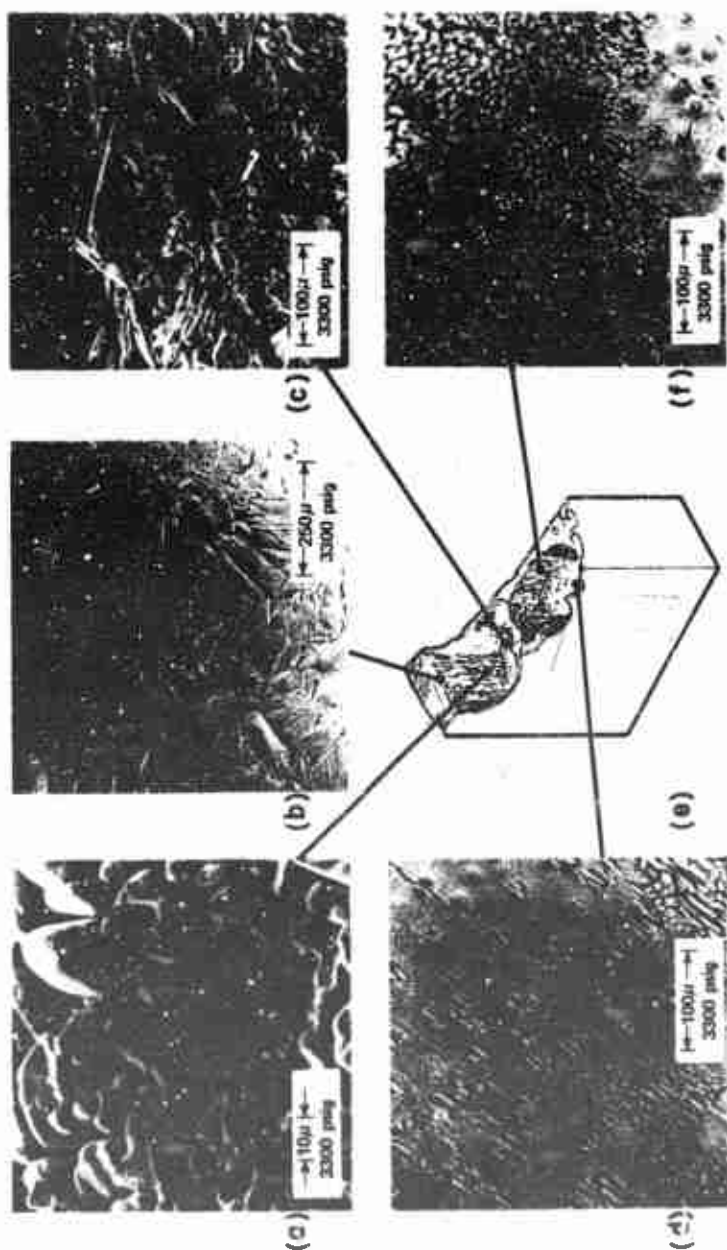


FIG. 3.16. Surface Morphology of Ammonium Perchlorate Samples Which Were Thermally Quenched While Self-deflagrating in a 3300 psig Nitrogen Atmosphere.

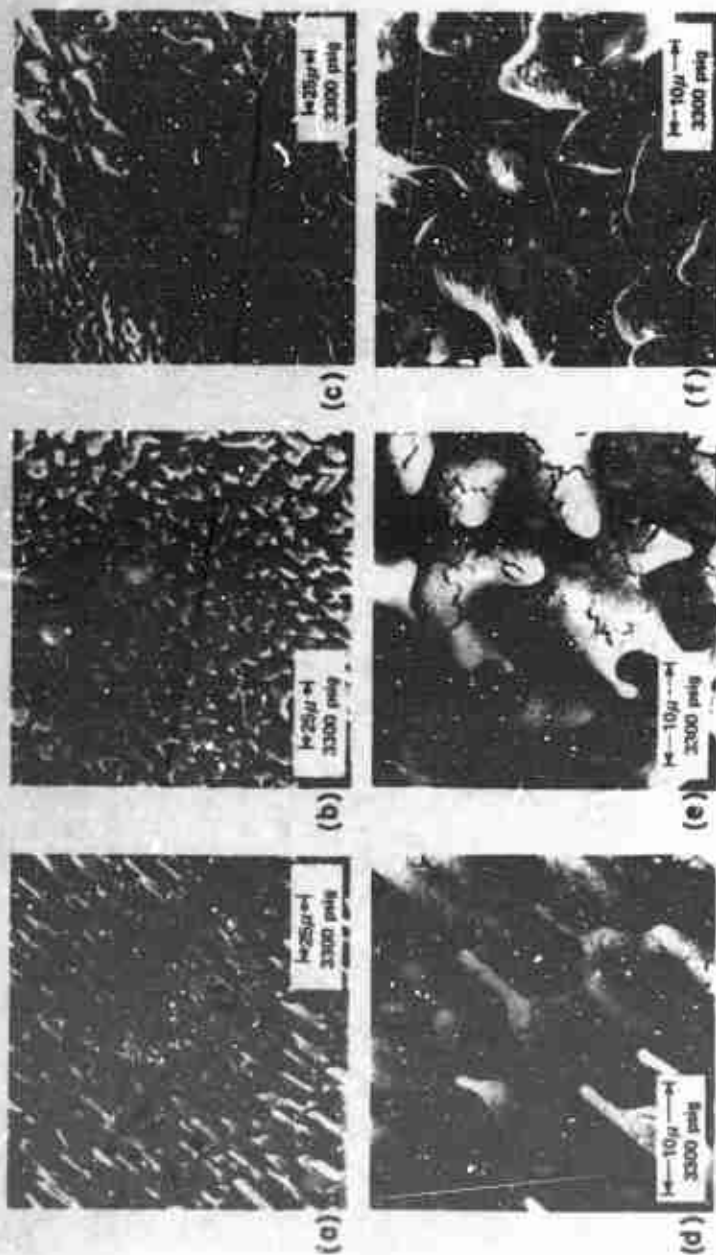


FIG. 3.17. Surface Morphology of Ammonium Perchlorate Samples Which Were Thermally Quenched While Self-deflagrating in a 3300 psig Nitrogen Atmosphere.



FIG. 3.18. Profile of Regressing Surface of Ammonium Perchlorate Single Crystal Undergoing Self-deflagration in the Pressure Regime 2000 $< p < 4000$ psi.

In the first profile the surface ignited in such a way that a few areas were heated allowing the flames of the second profile to be established. These flames heated the adjacent areas and the flame spread as indicated in the third profile. It should be noted that the context of profile here is not one of consecutive frames on the film, rather an interval of film is represented by consecutive profiles, so the fifth profile shows the effect of the flame spread and enhanced regression during the time interval since the fourth profile. The sequence is: (1) a portion of the surface is heated, (2) flame spreads to heated area, (3) enhanced regression rate ensues due to flame, (4) flame heats adjacent areas, and (5) the cycle repeats. The result is the pulsating regression and flame hopping observed in the motion pictures. This dependency of flame spread on the heating of an adjacent area may help explain why difficulty was encountered in the ignition and sustenance of self-deflagration for small samples, and why the sample size would be a parameter affecting the magnitude of the deflagration rate curve. Bobolev, et al (Ref. 7), found that the surface temperature measurements were characterized by a periodic change in temperature with a period of 50 msec and an amplitude of 500°C (1000°C - 500°C), and they also reported a pulsating flame.

3.7. SUMMARY OF SELF-DEFLAGRATION BEHAVIOR

The motion pictures of surfaces and flame behavior, the SEM pictures and the deflagration rate curve all indicate that the deflagration character of AP can be divided into at least three regimes for the pressures $300 < p < 4000$ psia and more probably four regimes for pressures between 300 and 10,000 psia. Table 3.1 summarizes the most important aspects of the deflagration of AP.

TABLE 3.1. Summary of Deflagration
Behavior of Ammonium Perchlorate

Regime	Pressure, psia	Rate, in/sec	Regression	Surface		Deflagration rate as a function of pressure
				Characteristic	as a function of pressure increase	
I	300- 900	0.13-0.34	steady planar	Gas entrapped in liquid resulting in a froth	Thickness of liquid layer decreases	$r = ap^n$ with $n = 0.8$
II	1000-2000	0.34-0.48	steady macroscopi- cally planar	Ridges and valleys with activity sites in valleys, pattern of surface spatial- ly invariant w/time	Length of ridges de- crease; activity sites go from froth → shallow depres- sions, and pillars → vigorous gas reac- tion within melt which expels liquid	Decreasing positive dr/dp with $n + 0$
III	2000-4000	0.48-0.10	pulsating nonplanar	Needles in areas of maximum regression	To be investigated	Negative dr/dp
IV	>4000	>0.10	To be in- vestigated	To be investigated	To be investigated	Positive dr/dp

4. LOW PRESSURE DEFLAGRATION OF AMMONIUM PERCHLORATE IN METHANE

This study was undertaken to ascertain if the low pressure self-deflagration limit was due to the inability of AP to support a liquid at pressures below approximately 275 psia. Single crystals of pure AP were deflagrated in methane atmospheres ranging from 15 psia to 200 psia and thermally quenched using the copper vice depicted in Fig. 3.3. The thermally quenched samples were examined with the SEM.

The surface of the sample deflagrated and quenched in the 200 psia methane atmosphere is depicted in Fig. 4.1a-d. The surface exhibits the structure previously seen to be characteristic of a melt through which gas was evolved. The thickness of the bubble wall (Fig. 4.1c) indicates that the viscosity of the liquid was significantly higher at this pressure than it was at the pressure at which the AP would self-deflagrate. The structure shown in Fig. 4.1d has the appearance of a previously constricted liquid which had rapidly cooled by expansion after a very rapid heating, and is similar to the liquid which was spewed out when a hot wire was shoved into the crystal (see Section 2.3).

The structure of the surface of the sample quenched at 150 psia (Fig. 4.2a) was very similar to that of the 200 psia sample. The thickness of the bubble wall can be inferred from the layers seen in Fig. 4.2b. Evidence that a gas passed through the melt at pressures of 100 and 50 psia is shown in Fig. 4.3e-b and 4.4e-d.

Whereas the previous samples all seemed to give evidence of a highly viscous liquid on the surface, the sample quenched at 20 psia (Fig. 4.5a-c) exhibited different structure. Most of the surface was relatively planar and pock-marked as can be seen in the upper left hand corner of Fig. 4.5a. Of interest are the few areas of randomly oriented needles which appear on parts of the surface. High magnification reveals that the needles are hollow. This hollow, regularly shaped needle structure has been observed to be characteristic of the vapor phase condensing on a cool substrate.

These observations indicate that the low pressure self-deflagration limit (≈ 275 psia) is not due to a lack of liquid at pressures below this limit. In comparing those samples which were self-deflagrated above 275 psia with those burned in methane atmospheres below 275 psia, the self-deflagrated samples seemed to have a less viscous melt but with more gas entrapped within the melt. This indicates that the pseudo-condensed phase contributes to the energy required for the AP crystal to undergo self-deflagration, especially at low pressures when gas phase reactions are



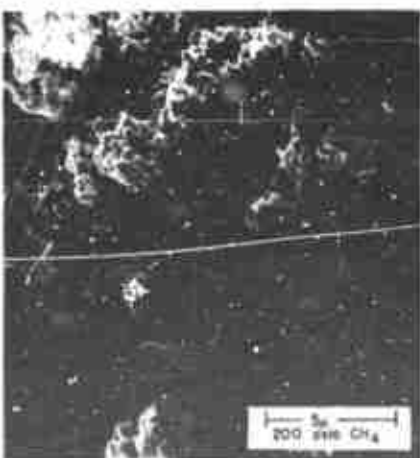
(a)



(b)



(c)



(d)

FIG. 4.1. Surface Morphology of Ammonium Perchlorate Samples Which Were Thermally Quenched While Deflagrating in a 200 psia Methane Atmosphere.

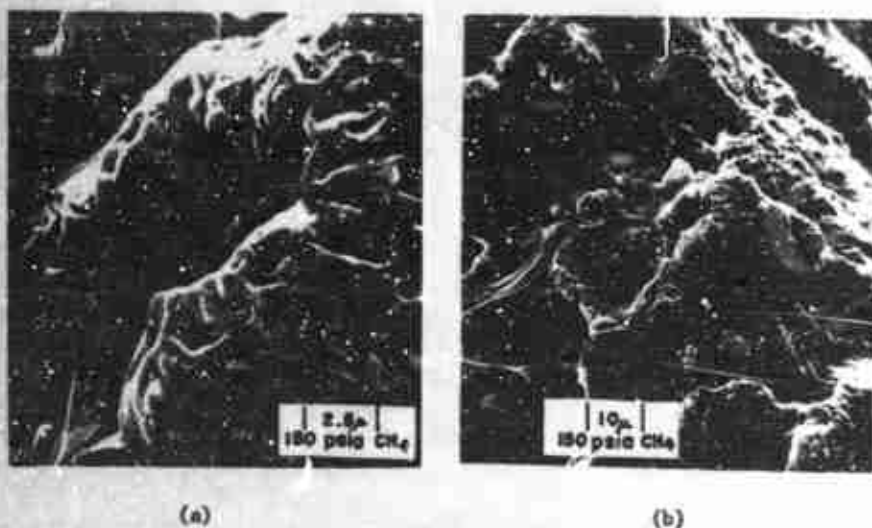


FIG. 4.2. Surface Morphology of Ammonium Perchlorate Samples Which Were Thermally Quenched While Deflagrating in a 150 psia Methane Atmosphere.

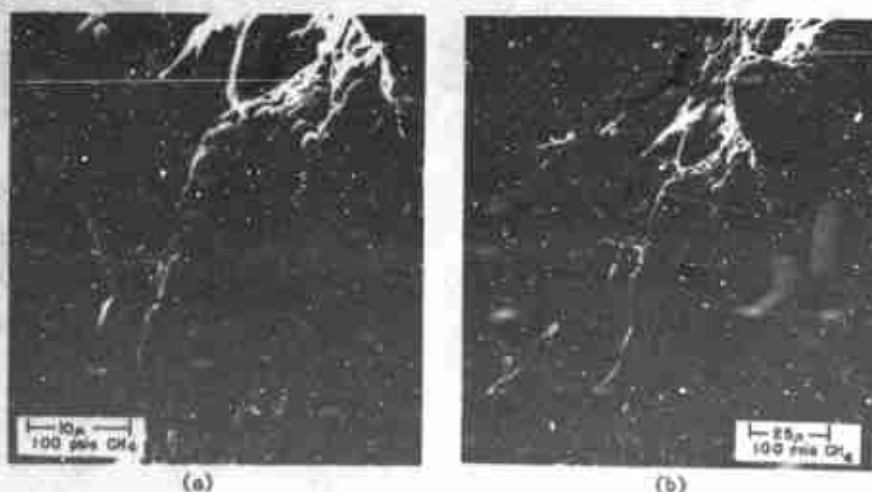
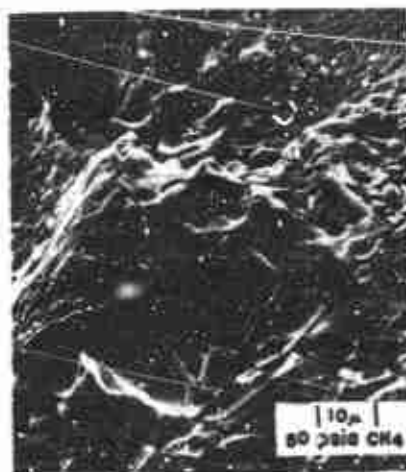


FIG. 4.3. Surface Morphology of Ammonium Perchlorate Samples Which Were Thermally Quenched While Deflagrating in a 100 psia Methane Atmosphere.



(a)



(b)

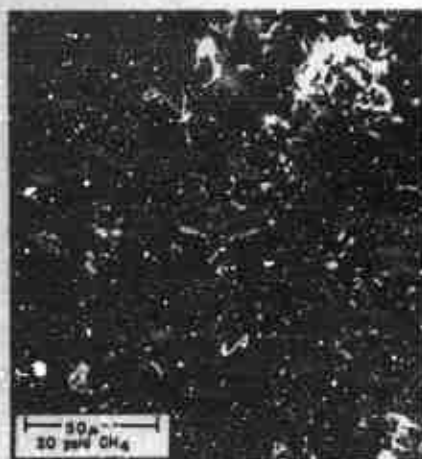


(c)



(d)

FIG. 4.4. Surface Morphology of Ammonium Perchlorate Samples Which Were Thermally Quenched While Deflagrating in a 50 psia Methane Atmosphere.



(a)



(b)



(c)



(d)

FIG. 4.5. Surface Morphology of Ammonium Perchlorate Samples Which Were Thermally Quenched While Deflagrating in a 20 psia Methane Atmosphere.

"fa-" from the crystal surface. The conclusion that the energy transfer rate is enhanced by the presence of a gas entrapping liquid is partially supported by the surface (see Fig. 4.6) of a thermally quenched sample deflagrated and quenched in a 15 psia methane atmosphere. The letters in Fig. 4.6 indicate regions where differing structural patterns were evidenced. Region A, the area of maximum regression, possessed a structure illustrated by Fig. 4.7. Such structure conforms to the observation made by Hightower (Ref. 14) on samples quenched by flooding the test chamber with nitrogen gas. Region B (Fig. 4.8a-c), the area of minimum regression, is characterized by regular shaped holes similar to the decomposition sites on an m-face of the AP crystal (Ref. 2) both in structure and arrangement on the sample surface. The results for Zone C are pictured in Fig. 4.9a-b. Of interest is the structure of the wall of the hole (Fig. 4.9b). The Area D, where maximum regression perpendicular to the direction of gross regression takes place, possesses a frothy appearance (Fig. 4.10a-d), associated with high heating rates (see Section 2.3).

The results of this study indicate that a liquid existed on AP samples that were deflagrated below 275 psia. This proves that the low pressure self-deflagration limit is not due to the inability of AP to form a surface melt at pressures below this limit.

It is felt that the use of a combustible atmosphere is an experimental complication that should be eliminated. Therefore, future investigation will utilize a CO₂ gas laser to supply the additional energy required to burn the samples, thereby eliminating flame spread down the sample sides and other artifacts due to uneven heating by the combustion reaction. An additional advantage of this method is that a quench can be obtained simply by terminating the additional energy flux.

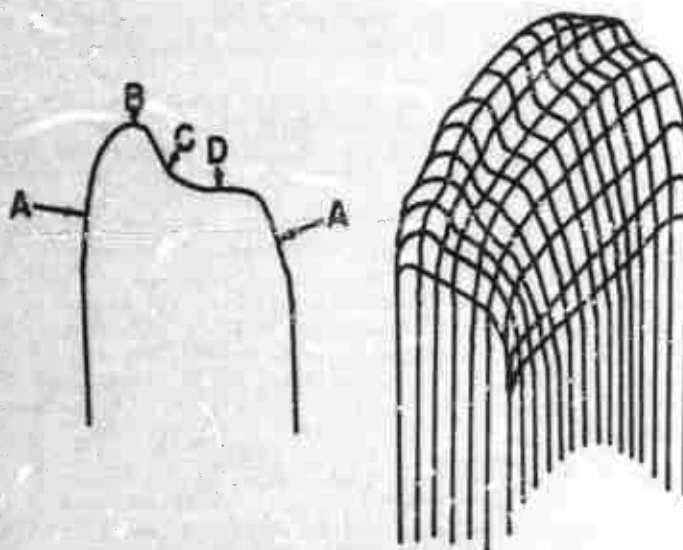


FIG. 4.6. Profile of Ammonium Perchlorate Sample Quenched at 15 psia Methane. The letters designate areas discussed in text.



FIG. 4.7. Surface Morphology of Area A of Ammonium Perchlorate Sample Quenched in 15 psia Methane.



(a)

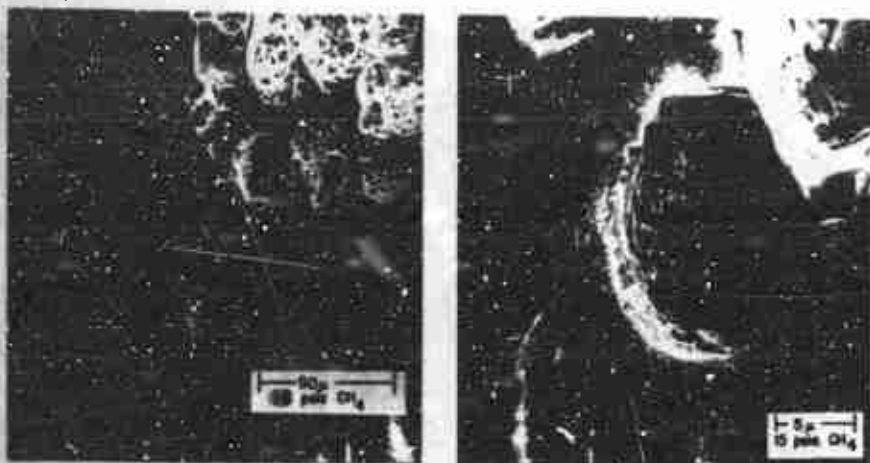


(b)



(c)

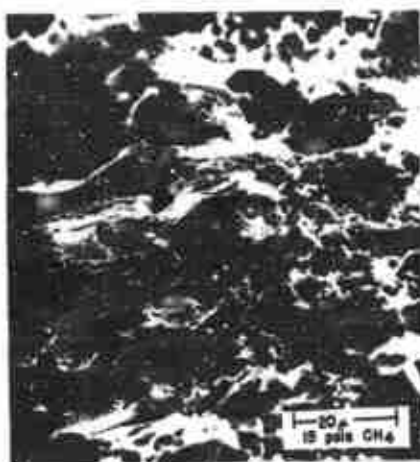
FIG. 4.8. Surface Morphology of Area B of Ammonium Perchlorate Sample Quenched in 15 psia Methane.



(a)

(b)

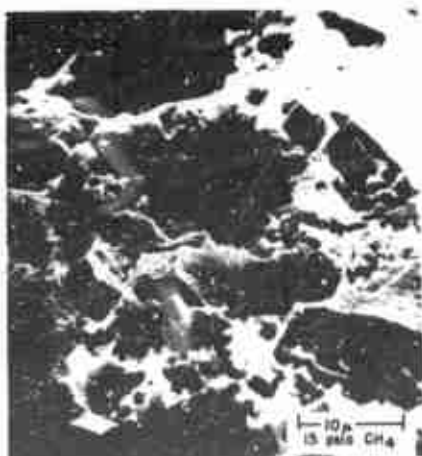
FIG. 4.9. Surface Morphology of Area C of Ammonium Perchlorate Sample Quenched in 15 psia Methane.



(a)



(b)



(c)



(d)

FIG. 4.10. Surface Morphology of Area D of Ammonium Perchlorate Sample Quenched in 15 psia Methane.

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13. ABSTRACT This report contains studies of the following behavior of pure single crystals of ammonium perchlorate: decomposition in vacuum, high heating rate decomposition at atmospheric pressure, self-deflagration in nitrogen atmospheres of 275 < p < 5000 psia and deflagration in methane atmospheres of 15 < p < 200 psia.			

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14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Oxidizers Ammonium perchlorate Decomposition Deflagration Self-deflagration Interface decomposition Deflagration rate Scanning electron microscope						

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